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Characterization of LPPS processes under various spray conditions for potential applications

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Abstract

Low Pressure Plasma Spraying (LPPS) is nowadays a well-established thermal spray process with a broad variety of important applications for functional surface coatings. The operating pressure for LPPS processes can vary in a wide range from typically 200 mbar down to only a few mbar. This leads to unconventional properties of the plasma jet, in terms of supersonic flow with strong shock structure at moderate pressure, towards rarefaction and frozen flow at very low pressure.

In order to optimize and control the spray processes for specific applications, it is necessary to understand the underlying physical mechanisms. However, so far only limited knowledge has been established on the plasma jet properties and its interaction with the spray particles in LPPS conditions.
We present several experimental investigations to characterize plasma spray processes under various pressure conditions. Measured plasma jet properties using a dedicated enthalpy probe system and imaging are combined with IR-pyrometry and velocimetry on the particles (DPV2000) to further improve the understanding of the plasma particle interactions. These results, along with spray deposit characterization, can be used to optimize the coating properties and explore further potential applications.

Introduction

In recent years the production of LPPS coatings has been successfully introduced into industrial markets for aeronautics, gas turbine, medical and other special applications. Especially in the area of gas turbine coatings for resistance to hot gas corrosion, the efficiency and reliability of the LPPS process for high quality coatings has provided its leading position.

With the new development of the LPPS Thin-Film technology (LPPS-TF), which is a special modification of conventional LPPS using reduced chamber pressures below 10 mbar, a new window has been opened to deposit uniform and dense thin layers onto large areas in short coating times. This spray process will allow the access to new market areas and will be able to bridge the gap between conventional thin film (< 1-10 μm) deposition using PVD/CVD processes and thick (> 50 -100 μm) thermally sprayed layers [1].

The LPPS technology is a development in thermal spray processes under controlled atmosphere, which allows coating under various low pressure conditions, typically ranging from 50 to 200 mbar [2]. In contrast to Atmospheric Plasma Spraying (APS) these processes have the advantage to allow for a controlled composition of the operating atmosphere in the chamber, to avoid oxidation or contamination of the powders and sprayed deposits. Additional features of LPPS are the application of a superimposed transferred arc mechanism and preheating of the substrate with the plasma jet. The variation of the operating pressure has also a strong influence on the length and diameter of the plasma which has an impact on the spray particle conditions (velocity, spray spot size). This allows for optimizing the particle spray plume for a given coating.

Previous investigations [3] on a laboratory DC argon plasma jet have shown the peculiarities of these plasma flows, in particular the supersonic flow structure with shocks and the reduced collisionality due to exhaust jet rarefaction at very low pressure. Since such operating conditions are completely different from those of APS, investigations of both the plasma jet and spray particle properties are necessary to acquire a basic understanding of this new process. This will allow for optimizing spray parameters and performances of the considered coatings as well as the investigation of new potential applications.

The LPPS system described here is an evolution of conventional LPPS equipment, specially designed for the development of thin and dense thermal coatings. First measurements with a dedicated enthalpy probe system have already been reported on this system using an Ar/H_2 plasma jet [4]. These data were interpreted assuming Local Thermal Equilibrium (LTE) [5].

Preliminary particle velocity measurements with a standard DPV-2000 diagnostic system have already been reported [6] in LPPS conditions, but without any information on the plasma jet properties.

In this paper, results using a modified DPV-2000 diagnostic system, of spray particle temperature and velocity under various LPPS operating conditions, down to the lowest pressure range, are presented for the first time. These results are combined with data of plasma jet properties obtained with enthalpy probe measurements on the same system and in the same conditions.

Experimental set-up

LPPS chamber and plasma gun

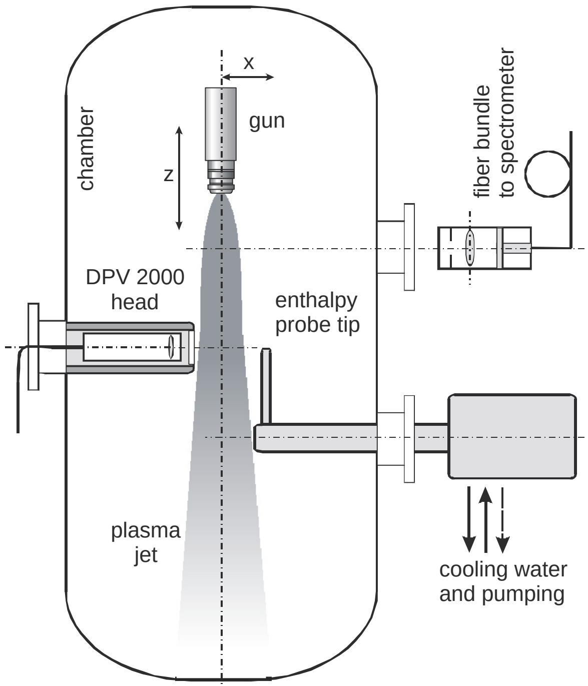


Figure 1: Schematic experimental set-up of enthalpy probe, spectrometer and DPV-2000 on the LPPS chamber.

The LPPS system described here is a specially designed R&D tool, equipped with a linear sting and a single load lock chamber for sample manipulation. The large vertical spray chamber allows for the increase of the plasma jet length to up to 2.5 meters and is equipped with several flanges and throughputs for the installation of control and diagnostic tools. Additional vacuum pumps enable to cover the whole operation conditions from standard LPPS, down to the pressure range of a few mbar. The plasma gun is a standard Sulzer Metco O3CP, single cathode, torch which can be operated down to 1 mbar with a total gas flow of up to about 150 SLPM. Depending on the nozzle design the injection from up to 4 powder feed lines is possible. The use of a tungsten insert in the copper anode allows operating the torch with electrical currents exceeding 2.5 kA which lead to input powers well above 100 kW.

A schematic arrangement of the gun inside the LPPS chamber is shown in Fig. 1, along with the installed plasma jet and particle diagnostics. The axial and radial positions of the plasma jet can be adjusted by linear movement of the gun along the torch axis (z-position) or perpendicular (x-position). Depending on the operating conditions the spray distances can vary from 150 to 1350 mm.

Plasma jet diagnostics

The enthalpy probe technique offers a relatively simple method for simultaneously measuring multiple plasma parameters and is nowadays extensively used for the diagnostics of thermal plasma jets in APS and VPS conditions. In order to characterize the plasma jet throughout the whole pressure range, various dedicated enthalpy probe tips have been used which allow to keep a good relative spatial resolution and a sufficient sample gas flow. The probe is fixed and the gun is moved to obtain radial and axial profiles of plasma enthalpy, density, temperature and velocity. Further details on the design of the enthalpy probe system which allows measurements at low pressure are presented elsewhere [4], along with the numerical technique used to infer the free-stream plasma jet properties from the stagnation measurements at the probe tip [3].

Spray particle diagnostics

We used a commercial DPV-2000 system from TECNAR, which has been modified to allow particle measurements under low pressure conditions. The underlying principle of this system is described in details elsewhere [7]. It allows the simultaneous measurement of particle temperature and velocity as well as particle flow rate and relative particle diameter. Particle velocity is determined by a time-of-flight technique during the travel of the individual particle images in front of a two slit mask. The particle temperature is inferred from the ratio of the thermal radiation at two selected wavelengths after proper calibration (two-color pyrometry). The dimensions of the optical mask and the wavelengths of the band-pass filters have to be chosen according to the spray conditions under investigation.

In LPPS conditions, specific band-pass filters have to be selected, because the plasma radiation is superimposed to the thermal particle radiation (extended plasma jet length). For this purpose, an optical spectrometer was used to detect the plasma radiation through an optical fiber bundle. The spectral range of 400 – 1000 nm was investigated under various spray conditions with different plasma gases and particle materials, and suitable filters were selected in spectral ranges free from plasma and particle vapor radiations.

The sensor head of the DPV-2000 was mounted in a specially designed, water cooled tube with a front optical window inside the vacuum chamber. This configuration allows accessing the axis of the plasma. However, the support tube structure cannot penetrate further inside the plasma because this could lead to perturbation of the plasma and particle jets and also results in excessive heat load on the tube. Therefore complete radial profiles have been obtained by scanning the gun along the y-axis (perpendicular to the x- and z-axis in Fig. 1).

Results

This chapter presents first results of the particle and plasma jet measurements taken at different LPPS operation conditions from 100 down to 1.5 mbar for Ar/He as well as Ar/H_2 plasmas. The injected powder used to measure particle properties is a 8% yttria stabilized zirconia of a suitable size distribution (-22+5μm) injected from a single feed line with 30 g/min. for all the spray conditions described here.

Standard LPPS conditions

Here 'standard operation condition' refers to as the typical pressure range under which LPPS or VPS processes take place which is around 50 - 200 mbar. Under such a reduced pressure the plasma jet is already clearly expanded and gives rise to enhanced plasma and particle velocities as compared to APS conditions. An Ar/He plasma (50/110 SLPM) has been investigated here at 100 mbar, as it is typically used with the LPPS process for a special application to obtain very dense and homogenous coatings with high deposition rates. The influence of the electrical input power on the plasma jet and particle properties has been investigated for different currents of 1500, 2000 and 2600 A, respectively. This corresponds to input power levels of 65 - 125 kW.

Axial profiles have been measured as close as possible to the nozzle exit without the risk of damaging the enthalpy probe or the DPV sensor tube due the exposure to high heat loads. The dependence of the axial particle properties on the distance from nozzle exit is shown in Fig. 2 for the different currents. Both particle velocity (Fig. 2a) and temperature (Fig. 2b) are increasing with current over the whole range due to the larger amount of input power. Maximum velocities on gun axis are observed between 500 and 600 m/s and stay well above 400 m/s up to distances of about 550 mm. They show a similar behavior for all currents with a clear maximum around 250 mm distance and a nearly linear decrease downstream due to the spreading and slowing down of the plasma jet. The velocity decrease towards the gun nozzle indicates the end of the acceleration zone of the particles which is more or less of similar length for all currents.

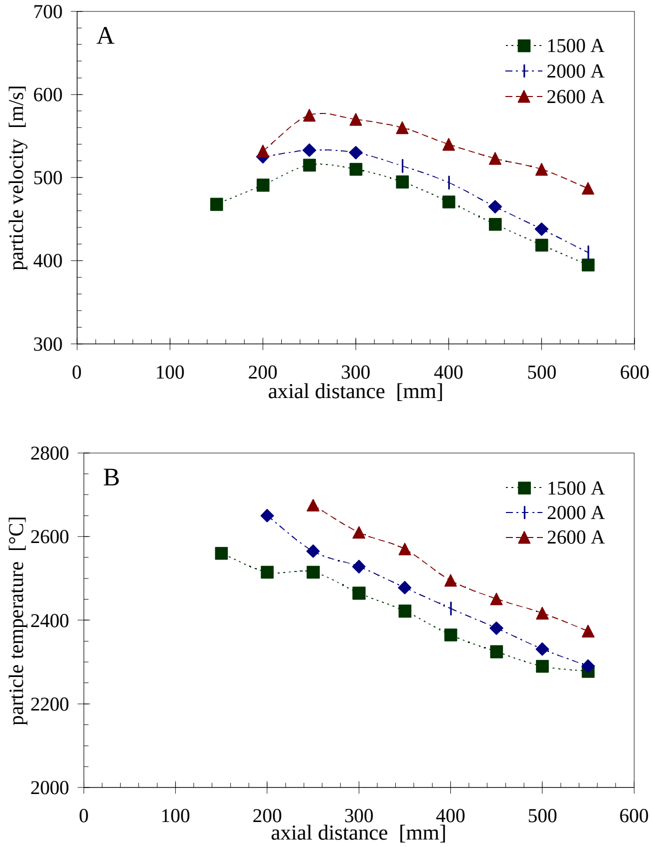


Figure 2: Axial profiles of the particle velocity (a) and temperature (b) for 3 torch currents (1500, 2000 and 2600A) at 100 mbar chamber pressure (50/110 SLPM Ar/He).

Maximum particle temperatures are observed to be around or slightly above the melting temperature of the powder (around 2600 °C). They show a linear decrease over the whole range investigated beyond 250 mm distance.

Radial profiles of the particle velocity and temperature, at 300 mm distance from nozzle exit, are shown in Fig. 3 for the three currents. Even if the powder is asymmetrically injected by a single injector, both velocity and temperature show relatively symmetric radial profiles at high current. With decreasing current the profiles are more influenced by the injection direction and are slightly off-axis in the opposite direction of the injection. The profiles broaden slightly with increasing current. The particle jet width can be determined by the particle detection rate which is below 2 particles/s at the edge of the profile. This leads to a reduction of the particle jet diameter from about 150 to 120 mm when the current is decreased from 2600 down to 1500 A, which is consistent with the diameters of the corresponding spray patterns observed under these conditions. Peak values decrease with reduced torch current from 570 to 510 m/s and from 2580 to 2500 °C, respectively. Even at the edge of the particle beam both velocity and temperature are still at levels above 300 m/s and 2100 °C, respectively, indicating a relatively homogenous distribution of the particle properties over the whole spray pattern.

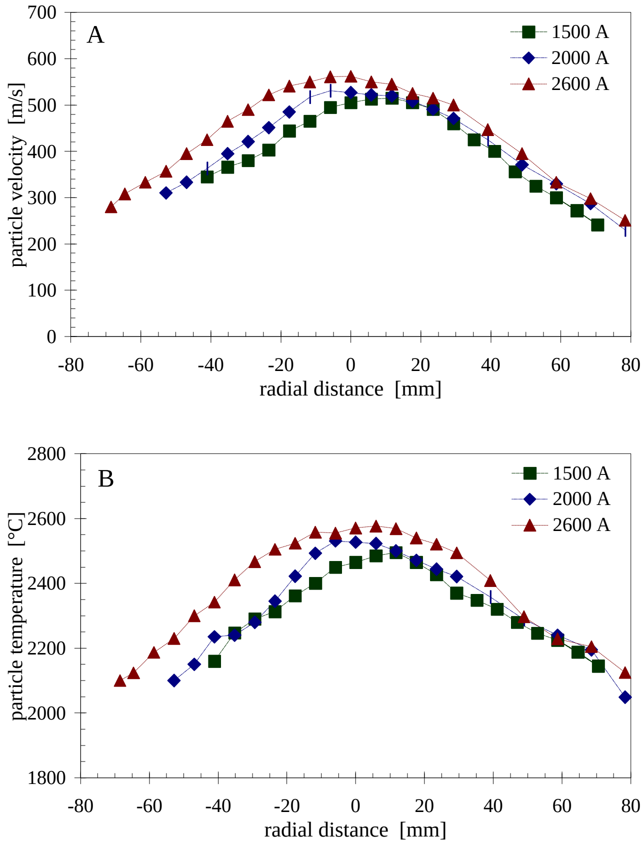


Figure 3: Radial profiles at 300 mm axial distance, of the particle velocity (a) and temperature (b) for the same conditions as in Fig. 2.

The corresponding enthalpy probe results of plasma velocity and temperature are shown, for the same spraying conditions, as a function of the axial distance in Fig. 4 and radial profiles at z = 300 mm in Fig. 5. The temperature and velocity of the jet increase strongly towards the nozzle for distances less than 200 mm at 1500 A. This goes along with a change of the slope at this distance which corresponds to the section where the plasma jet slows down and spreads out due to cold surrounding gas engulfment and turbulence. Note that this location corresponds to the end of the particle acceleration zone in Fig. 2a, because the jet velocity is no longer sufficient to accelerate them further.

At 2600 A, due to a high total heat load (above 6000 W) onto the probe, measurements had to be limited to distances beyond 250 mm, where plasma temperature is already above 6000 K and specific enthalpy around 8000 kJ/kg. In almost the whole region investigated, the plasma flow is compressible and subsonic (Mach numbers between 0.3 and 1). The current has less effect on the plasma enthalpy or temperature than on the jet velocity and length. This explains its influence on the particle properties which is more pronounced for velocity due to momentum transfer than for temperature by heat transfer.

Note that the particle parameter profiles are more peaked than the profiles of the plasma parameters at the same location. This is due to the fact that the particle properties at a given axial location depend on the whole history of their interaction with the plasma during their flight upstream of this location. And it is known that the plasma properties are more peaked upstream, in the supersonic region close to the nozzle [3].

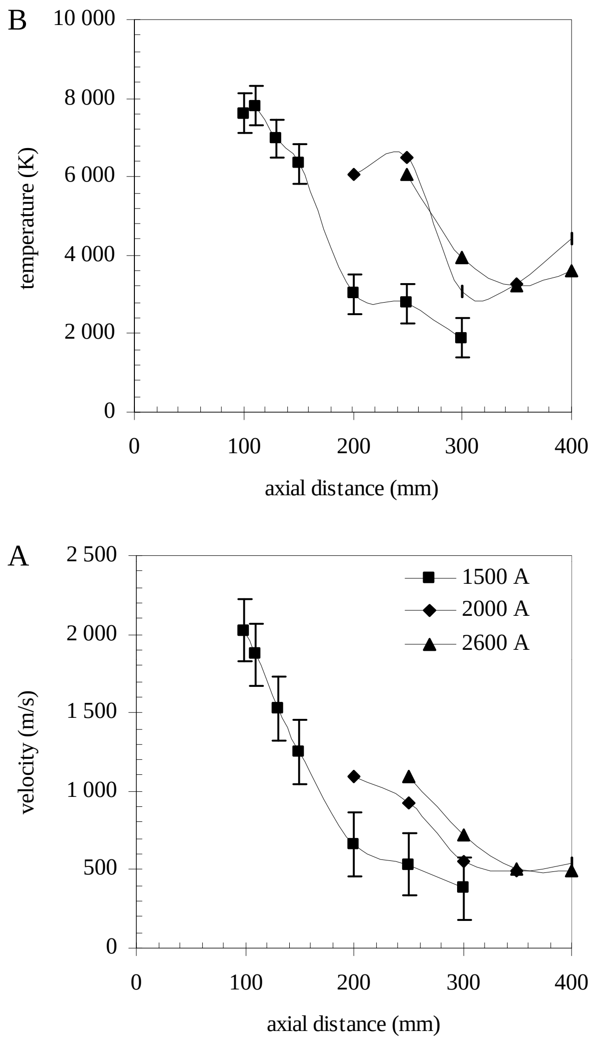
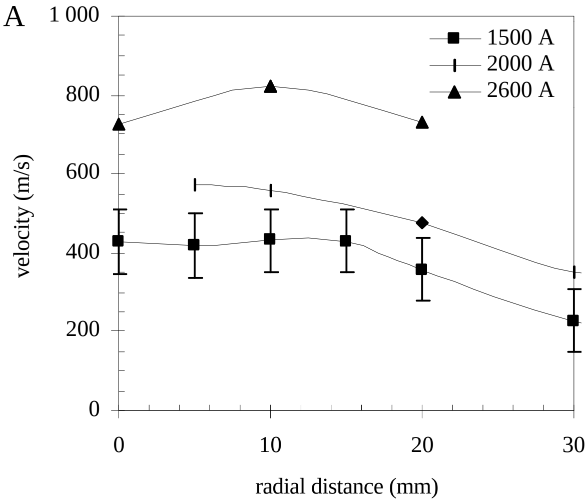


Figure 4: Axial profiles of the plasma velocity (a) and temperature (b) for the same conditions as for Fig. 2.

A



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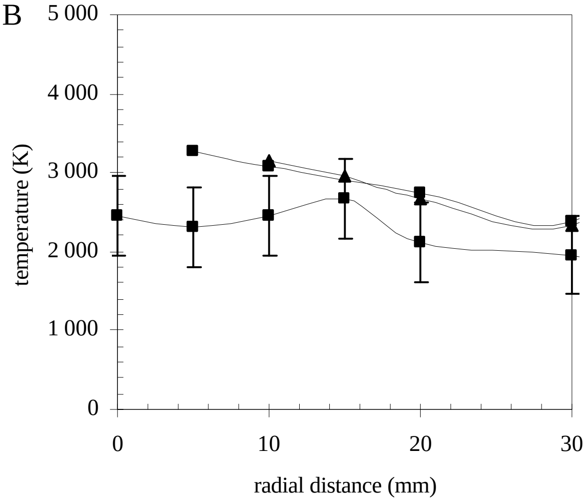

Figure 5: Radial profiles at 300 mm distance from the nozzle exit, of the plasma velocity (a) and temperature (b) for the same conditions as for Fig. 2.

LPPS Thin Film conditions

Operation under chamber pressures down to 1 - 2 mbar allows for applying coatings in the 5 - 50 µm thickness range onto large areas within a short deposition time. This new plasma spraying regime (LPPS-Thin Film) is characterized by a strong enlargement of the plasma plume in length (more than 2 m) and diameter (about 200 - 400 mm). An image of a typical LPPS-TF plasma jet operated with Ar /H_2 is shown in Fig. 6 with a 20 mm diameter enthalpy probe inside which give an idea of the dimensions. The main characteristics of this kind of plasma jets are: supersonic flow with weak turbulence, moderate local heat flux and deviations from the Local Thermodynamic Equilibrium (LTE) due to rarefaction effects [3]. Operation with either Ar/He or Ar/H_2 gas compositions leads to a completely different behavior of the plasma jet as known from traditional APS and LPPS conditions. The high viscosity of helium leads to a more laminar plasma flow with enhanced momentum transfer to the particles, whereas the dissociation of hydrogen leads to an increased specific enthalpy and a consecutive high heat transfer to the powder.

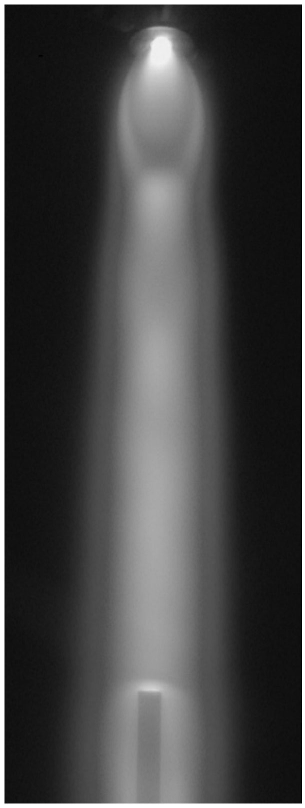

Figure 6: Typical view of a LPPS-Thin Film plasma jet in Ar/H_2 with a 20 mm diameter enthalpy probe tip inside.

LPPS-Thin Film Ar/He-plasmas

Figure 7 shows the particle properties of the YSZ powder sprayed with an Ar/He plasma (50/110 SLPM, 1500A) at 100 mbar and at 1.5 mbar chamber pressure for comparison. These radial profiles have been taken at spray distances of 300 and 450 mm, respectively. Clearly seen is the flattening of both velocity and temperature profiles at 1.5 mbar. Hence, even at a comparable spray distance of 450 mm for 1.5 mbar, the width of the spray pattern is markedly enlarged compared to the 100 mbar conditions. This effect is further pronounced with increasing spray distance.

The peak values on gun axis are significantly lower at 1.5 mbar (300 - 350 m/s and 1900 - 1950 °C, respectively). Even if the measured particle temperature is far below the melting point of the powders, dense coatings can be obtained in these conditions, which is still beyond our understanding. An explanation could be that the layer is mainly build by small molten particles < 12 µm which are below the detection limit of the DPV 2000. The very fine microstructure of these coatings gives evidence to this assumption. The powder deposition efficiency strongly depends on the powder size.

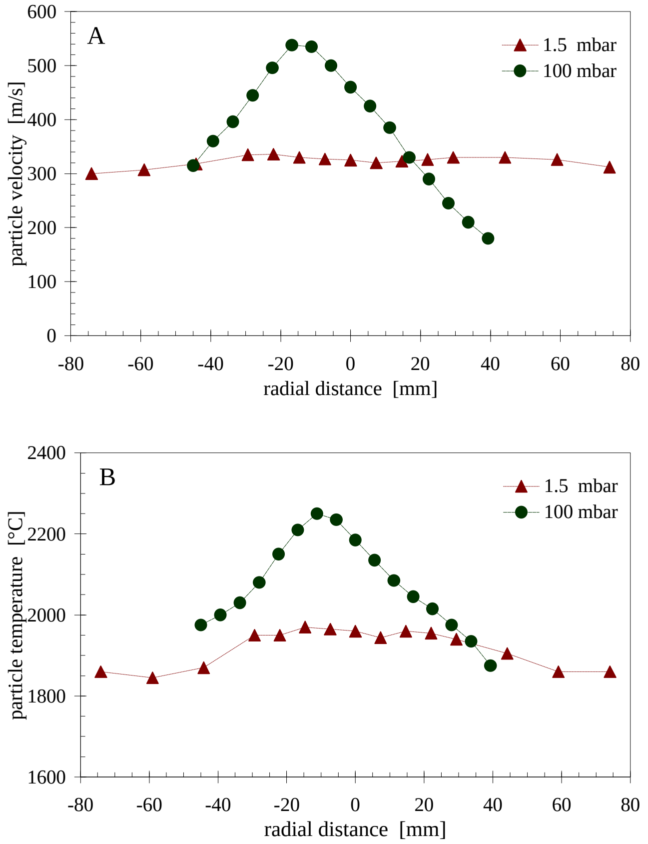

Figure 7: Radial profiles of the particle velocity (a) and temperature (b) for an Ar/He (50/110 SLPM) plasma at 1500 A and for two different pressures (100 and 1.5 mbar). Axial distance is 300 mm at 100 mbar and 450 mm at 1.5 mbar.

The flattening of the jet velocity and temperature profiles at low pressure is also observed with the enthalpy probe as shown in Fig. 8 at 400 and 800 mm distance from the nozzle exit. Along the axis the plasma temperature is quite constant and the velocity slightly decreases over the distance investigated. This is because the flow is very laminar and well isolated from the surrounding cold gas. In addition, the low plasma and gas density result in weak collisionality which further reduce the interactions of the jet with the surroundings.

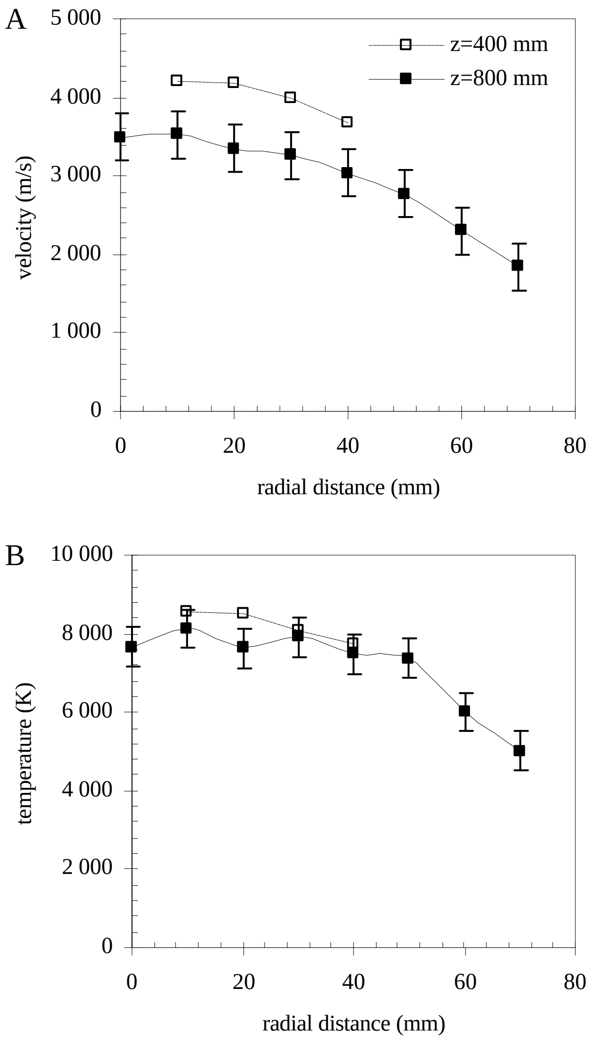

Figure 8: Radial profiles of the jet velocity (a) and temperature (b) taken at 2 axial distances for 1.5 mbar. Same plasma conditions as for Fig. 7.

<u>LPPS-Thin Film Ar/H_2 Plasmas</u>

A typical Ar/H_2 plasma with 100/3 SLPM in the pressure range of 2 - 10 mbar has been investigated at a torch current of 1500 A, corresponding to an input power of 70 kW. Radial profiles at 775 mm from the nozzle exit as well as axial profiles for three different chamber pressures (2, 6 and 10 mbar) have already been measured with enthalpy probe and described in a previous paper [4]. These data have been recalculated using an improved analysis [3]. The overall behavior of the plasma properties remains unchanged, only absolute values turned out to be reduced by around 10 - 15% for velocity and 25 - 35% for temperature, respectively.

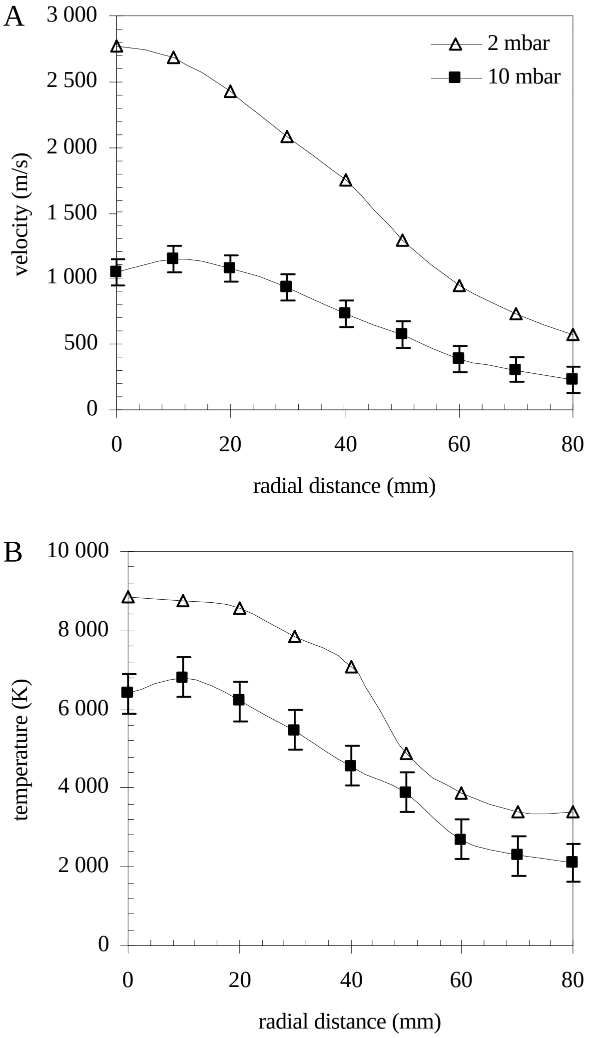

Figure 9: Radial profiles of the jet velocity (a) and temperature (b) taken at an axial distance of 775 mm for 10 and 2 mbar. Plasma conditions: 1500 A, 100/3 SLPM Ar /H_2.

For the 2 mbar case, the axial profiles have been extended from 600 down to 250 mm distance from the gun nozzle. Both plasma velocity and temperature are fairly constant along the axis showing only a slight increase towards the nozzle (typically 3000 - 3400 m/s and 8500 - 9500 K, respectively). In contrast, the local heat flux increases from 4 up to 6.5 MW/m^2, because the density is increasing.

A significant increase in plasma velocity, temperature and free stream enthalpy is observed as the pressure is reduced, as shown in Fig. 9. However, the mass density decreases by almost one order of magnitude as the pressure is reduced from 10 mbar down to 2 mbar. A consequence is that the local heat fluxes are nearly comparable for these 2 pressures as shown in Fig. 10. This fact explains why the particle properties are not so different for these two pressures (see below).

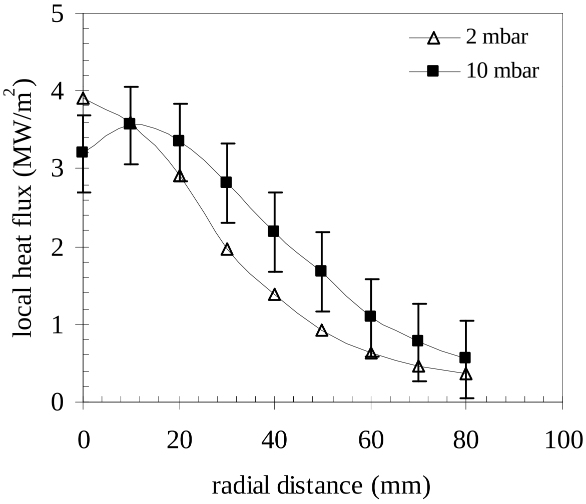

Figure 10: Radial profiles of the local heat flux for the same conditions as for Figure 9.

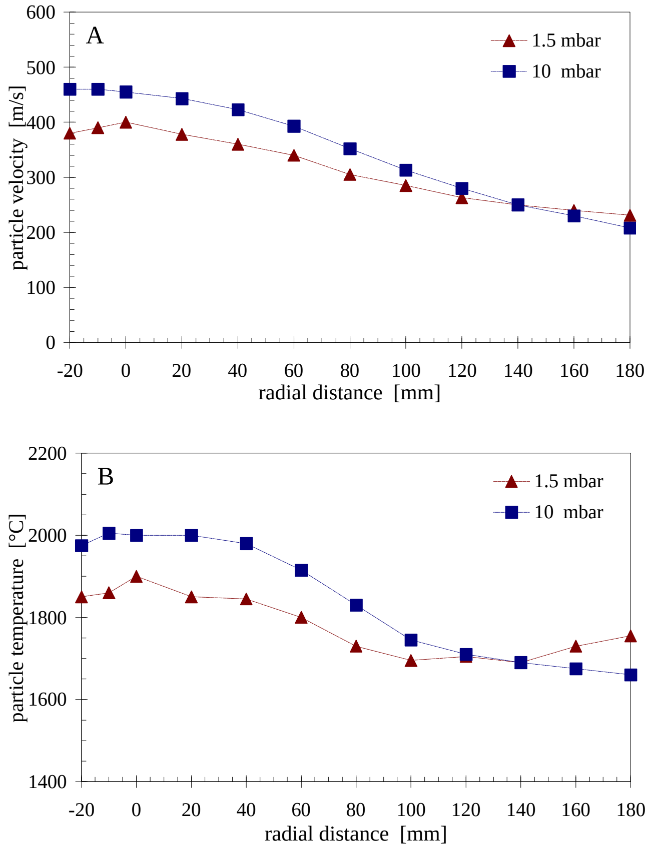

Figure 11: Radial profiles of the particle velocity (a) and temperature (b) taken at an axial distance of 975 mm for 10 and 2 mbar. Plasma conditions same as for Fig. 9.

Corresponding measurements of the particle properties with the DPV-2000 have been performed so far only at a spray distance of 975 mm. They are shown in Fig. 11 as radial profiles for particle temperature and velocity. Both only slightly increase with increasing pressure. At this spray distance the particle deposition profile extends to at least 300 mm in diameter. This will increase further to up to 400 mm at a longer spray distance of around 1350 mm. In Fig. 11 only half of a radial profile is shown since the measurements have been taken by moving the plasma gun towards the DPV sensor tube along the x-axis.

Summary and Conclusion

Experimental investigations of LPPS processes under various operation conditions have been performed using dedicated plasma jet and particle diagnostics. The results show the strong effect of the operating pressure on the plasma and particle properties. In standard LPPS conditions the radial profiles of the plasma and particle spray plume are broader and the velocities are fairly higher than in APS conditions. This gives rise to a relatively large deposition profile. At low current a clear asymmetric radial distribution due to single powder injection is evidenced. It is more symmetric for high current, where increased jet velocity and temperature result in a more efficient acceleration and heating of the injected powder. Comparisons between axial profiles of particle and plasma velocity show that the particle acceleration and deceleration zones are related to variations in the corresponding plasma velocity profile.

For the LPPS-Thin Film process, both plasma and particle properties are strongly modified with respect to the standard LPPS case: Particle temperature and velocity are significantly lower, whereas the plasma jet velocity and temperature are much higher than for standard LPPS. In addition the radial profiles are much flatter and axial variations are weak.

These measurements show preliminary correlations between particle and plasma properties and could eventually be used as input for modeling of the plasma-particle interactions (momentum and heat transfer). However, since particle properties at a given spray distance result from their history along the whole plasma jet, additional knowledge of the plasma properties would be required for axial distances closer to the nozzle. Such further investigations could then provide a more complete understanding of the basic mechanisms involved in these processes and finally help in the optimization of potential coating applications.

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